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ORIGINAL PAPER

A macromolecular *N*-bromosulphonamide as a heterogeneous oxidant in acidic media

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Abstract Styrene–divinylbenzene copolymer containing *N*-bromosulphonamide groups as sodium salt, [P]–SO_2NBrNa, can be viewed as a high molecular weight redox polymer mimicking its low molecular weight analogue: Bromamine-T. The stability of this copolymer in acidic media has not been studied in detail to date. This paper reports on studies of the reaction between the copolymer and aqueous $FeSO_4$ solution conducted in a series of batch and column experiments. It was found that pH <3.0 results in transition of active bromine from $–SO_2NBr^-$ groups to the solution, followed by oxidation of Fe^{2+} ions to Fe^{3+} ions by elemental bromine. In reaction media of pH >3.0, active bromine remains bound to the functional groups, facilitating application of this copolymer as an effective oxidant in column processes running in weakly acidic reaction media. Results of the present work provide evidence that $–SO_2NBr^-$ functional groups of the copolymer can stably exist in acidic form which is in contrast to the behaviour of the low molecular weight analogue (*N*-bromo-*p*-toluenesulphonic acid does not occur as a pure chemical compound).

Keywords Redox copolymer · Heterogeneous oxidant · Macromolecular · *N*-bromosulphonamide · Active bromine · Fe(II) oxidation

Introduction

Bromamine-T (BAT, sodium salt of *N*-bromo-*p*-toluenesulphonamide) is a low molecular weight organic compound with an oxidizing effect, often used in oxidation reactions of, among others, alcohols, aldehydes, ketones and amines

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[1–5]. Contemporary studies using BAT mainly concern the examination of drugs oxidation mechanisms in biological systems (drugs used, for instance, in the treatment of allergies and diabetes) [6–11]. Also, research work is conducted on the possibility of obtaining aziridine compounds from olefins (used, among other things, in the production of pharmaceuticals and optically active compounds) using BAT as a source of nitrogen [12–15]. The researchers dedicate a lot of attention to explanation of the oxidation reaction mechanism in acidic media, because under these conditions many reactions occurred much more favourably than without the addition of an acid or in the presence of alkaline medium. Under such conditions BAT (Ar-SO_2NBrNa) transforms to Ar-SO_2NBrH (1), which is unstable and undergoes reactions (2) and (3), as follows [3]:

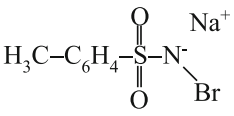
$$\mathrm{Ar-SO_2NBrNa} + \mathrm{H_3O^+} \leftrightarrow \mathrm{Ar-SO_2NBrH} + \mathrm{H_2O} + \mathrm{Na^+} \quad (1)$$

$$2\ \mathrm{Ar-SO_2NBrH} \leftrightarrow \mathrm{Ar-SO_2NH_2} + \mathrm{Ar-SO_2NBr_2} \quad (2)$$

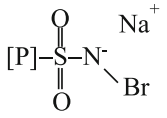
$$\mathrm{Ar-SO_2NBrH} + \mathrm{H_2O} \leftrightarrow \mathrm{Ar-SO_2NH_2} + \mathrm{HOBr} \quad (3)$$

Synthesis of high molecular weight, water-insoluble reagent containing functional groups of similar chemical composition and properties generates new possibilities for research in this field.

A macromolecular BAT analogue is a macroporous copolymer having a styrene–divinylbenzene skeleton containing *N*-bromosulphonamide functional groups in a sodium form (R/BrNa):



BAT



R/BrNa

[P] stands for the copolymer styrene–divinylbenzene macroporous structure.

We have described previously a method to obtain this copolymer, through a chemical modification of a commercially available sulphonic cation exchanger having a macroporous structure [16]. The product has a physical form analogous to ion exchangers, and its functional groups attached to the crosslinked macroporous matrix demonstrate high reactivity. Up to now, we have used materials of this type as heterogeneous oxidants to remove from water admixtures that are reductants. R/BrNa used in column processes allowed the removal, from water (through oxidation), of such burdensome or toxic admixtures as cyanides, thiocyanates, sulphides, and arsenites [17–20]. This copolymer was particularly useful for the oxidation reaction in basic media in which similar copolymers containing in their functional groups active chlorine atoms are poorly reactive or unstable [19, 21]. Thus, in the experiments conducted to date, we have primarily used R/BrNa, because of its pronounced reactivity in basic media. Up to now the behaviour of R/BrNa in acidic media has not been examined in detail. In our previous paper related to the removal of SCN^- ions

from solutions, it turned out that during the column process the pH value of media considerably decreased (down to a pH value of 1.7–1.8) and bromine separated from the bed, as a result of which the copolymer lost its oxidizing property [18]. This phenomenon can be observed visually by the disappearance of the yellowish colour of the copolymer and coloration of the effluent. This phenomenon has proved that at a certain, low pH value functional groups lose active bromine.

A question arises whether it is possible to use R/BrNa in acidic media, and what is the allowable, in approximation, low pH value of this oxidant's operation. The recognition of the properties and durability of R/BrNa in acidic media can broaden the scope of its application since it is precisely the low pH value that fosters many oxidation reactions. To answer this question, aqueous $FeSO_4$ solutions were selected as a substrate in the series of experiments studying the pH dependence of the following reaction:

$$[P]-SO_2NBrNa + 2\,Fe^{2+} + 2\,H^+ \rightarrow [P]-SO_2NH_2 + 2\,Fe^{3+} + Na^+ + Br^- \quad (4)$$

The aim of the present research was to examine the effect of the *N*-bromosulphonamide copolymer in the solution of a reductant the reaction of which is acidic. The point was to assess how low the pH value can drop while the heterogeneous oxidant still maintains in its functional groups the atoms of bromine, this being of utmost importance in the oxidation processes using the dynamic regime. Heterogeneous oxidizing agent, that is, stable and water-insoluble reactants, fit for use in a column process, providing a high redox potential, demonstrating a high oxidizing capacity, stable in terms of chemistry (i.e., consuming the oxidizing capacity gradually, as the reductant solution is supplied onto the column) can be helpful in resolving many environmental problems resulting from the presence in various types of water in a microamount of toxic admixtures that are reductants.

Experimental

Materials

The heterogeneous oxidant used in this study, a crosslinked styrene–divinylbenzene copolymer with *N*-bromosulphonamide groups (R/BrNa), was prepared by chemical modification of the commercially available cation exchanger *Amberlyst 15* (Rohm and Haas Co.). *Amberlyst 15* is a macroporous poly(S/20 %DVB) resin having $-SO_3H$ groups (4.70 mmol g^{-1} in the dry state); surface area 45 m^2 g^{-1}; average pore diameter 25 nm. Sulphonic groups of the resin were transformed into *N*-bromosulphonamide groups by a method published elsewhere [16]:

$$\begin{aligned} &[P]-SO_3H \xrightarrow{PCl_5/POCl_3} [P]-SO_2Cl \xrightarrow{NH_4OH} [P]-SO_2NH_2 \\ &[P]-SO_2NH_2 \xrightarrow{NaOCl} [P]-SO_2NClNa \xrightarrow{NaBr} [P]-SO_2NBrNa \end{aligned} \quad (5)$$

The product contained $-SO_2NBrNa$ groups 1.64 mmol g^{-1} (i.e., 3.28 mequiv of active bromine g^{-1}) and additionally $-SO_3Na$ groups 0.5 mmol g^{-1}. The presence

of sulphonic groups in the R/BrNa is a result of hydrolysis which occurs as a side reaction during the sulphonamide preparation.

Analytical grade ferrous sulphate (a reducing agent) was used for preparation of aqueous solutions. The solution used in the batch regime was 0.02 mol L^{-1} $FeSO_4$ (1117 mg Fe^{2+} L^{-1}) in deionized water. The solution used in the experiments carried out in the dynamic regime was 0.0025 mol L^{-1} $FeSO_4$ (139 mg Fe^{2+} L^{-1}) in deionized water.

Batch studies

All experiments were carried out at room temperature. In the experiments involving the batch regime, samples of the resin (0.61 g in the dry state, 1 mmol of active bromine) were contacted with 0.02 mol L^{-1} $FeSO_4$ solution in a different volume (200, 50 mL) ensuring a proper mole ratio—excess of ferrous ions or active bromine in relation to the stoichiometry of the reaction (4). Time-dependent measurements of Fe^{2+} and Fe^{3+} content in the solution were made. After 24 h, the polymeric reagent was separated and the content of Fe^{2+}, Fe^{3+}, and Br^- and pH was measured in the solution.

The redox titration of the resin was performed using 0.02 mol L^{-1} $FeSO_4$ solution. Eleven samples of the resin (0.61 g) were placed into separated vessels. Then the identified volumes of $FeSO_4$ solution were introduced, which required bringing about, e.g., (5) 50 % (50 mL), (9) 100 % (100 mL) reduction of the active bromine present in the functional groups of the R/BrNa. To sample (1) only deionized water was added. The closed vessels were protected from light and from contact with air and shaken at constant, room temperature [22]. After 24 h in the solution the redox potential and pH were measured and the content of Fe^{2+} and Fe^{3+} was determined.

Dynamic regime

In the experiments involving the dynamic regime, a sample of the resin (5.0 g in the dry state, $\sim$8.5 mL after swelling in water, 8.2 mmol of active bromine) was packed into a glass column. An aqueous solution of $FeSO_4$ (0.0025 mol L^{-1}) was passed through the resin bed. The flow rate was 29 BV h^{-1}. The samples of the effluent (250 mL) were collected and analysed for the content of Fe^{2+}, Fe^{3+}, and Br^-, and the pH was measured. After the process was finished (when the copolymer lost its oxidizing capacity) the resin bed was washed with 250 mL of 1 mol L^{-1} H_2SO_4, and the solution was analysed for the Fe^{2+} and Fe^{3+} content. Symbols used in the experiments: C is the concentration of Fe^{2+} in the effluent, V is the volume of effluent, C/C_0 is the changes in concentration of Fe^{2+} in the effluent, V/V_0 is the ratio of effluent volume to resin (bed) volume.

Chemical analyses

The active bromine in the copolymer was determined by the iodometric method. The ferrous and ferric ion concentrations were determined by standard

spectrophotometric methods (Spekol 1200, Analytic Jena, Germany). The Fe^{2+} ions concentration was determined using the formation of an orange complex compound with 1,10-phenanthroline monohydrate. The absorbance measurement was taken at the wavelength 510 nm. The Fe^{3+} ions concentration was determined using the formation of a red-coloured complex compound with potassium thiocyanate. The absorbance measurement was taken at the wavelength 480 nm. The redox potential was measured by means of the platinum/calomel electrode pair, the pH values were tested by means of the glass/calomel electrode pair, and bromide ions were estimated by argentometric titration using 0.01 mol L^{-1} $AgNO_3$ with the Ag/AgCl/calomel electrode system using a CPI-501 pH/ion meter, Elmetron, Poland.

Results and discussion

In the testing conducted using the batch regime solely a 0.02 mol L^{-1} solution of $FeSO_4$ in deionized water of pH 3.65 was used. This pH was neither corrected downwards with addition of an acid (due to the said low and insignificant durability of R/BrNa in acidic media), nor upwards with addition of a base (precipitation of $Fe(OH)_2$ from the solution would follow).

At the first stage of the testing the oxidizing capacity of the R/BrNa copolymer in relation to Fe^{2+} was determined. To this aim, reductant ($FeSO_4$) was contacted in a ratio providing 100 % of the reductant excess in relation to the amount resulting from the stoichiometry of the reaction (4). A sample of the R/BrNa copolymer containing 1.0 mmol of active bromine was treated with a 0.02 mol L^{-1} $FeSO_4$ solution containing 4 mmol of Fe^{2+}. During the course of the reaction the pale yellow solution turned yellow-orange. The characteristic smell of bromine was perceptible. It follows from the course of the curve presented in Fig. 1 that the oxidation reaction of Fe^{2+} ions occurred quickly; it was practically completed after 1 h.

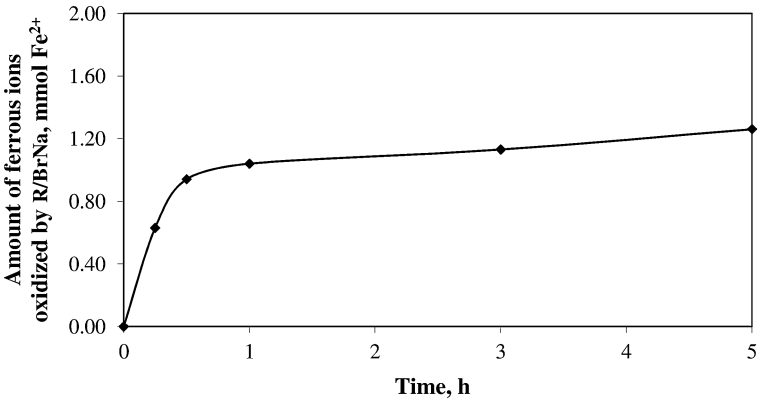


Fig. 1 Amount of ferrous ions oxidized by *N*-bromosulphonamide copolymer in solution in batch-wise reaction: 0.61 g R/BrNa + 200 mL 0.02 mol L^{-1} $FeSO_4$ in water

As follows from the data presented in Table 1, the reaction was accompanied by a drop of pH, from 3.65 to 2.91. In the post-reaction solution 1 mmol of Br^- was detected, i.e., an amount consistent with the content of active bromine in the copolymer sample before the reaction. The fact of a change in the colour of the reaction solution, the rapid course of the reaction, as well as the absence of active halogen in the sample upon completion of the process, demonstrate that the reaction could have an autocatalytic course, caused by the drop in pH. The change in the colour of the solution (which subsided after a certain time) evidenced that bromine separated from the copolymer that oxidized Fe^{2+} ions afterwards. During the reaction, the copolymer sample changed in colour from cream to brown. This was related to the separation of iron hydroxide within the copolymer structure:

$$[P]-SO_2NBrNa + 2\,FeSO_4 + 6\,H_2O \rightarrow [P]-SO_2NH_2\#2\,Fe(OH)_3 + NaBr + 2\,H_2SO_4 \quad (6)$$

means that $Fe(OH)_3$ has been dispersed in the sulphonamide copolymer matrix.

Upon completion of the reaction, the sample was washed off with 1 mol L^{-1} H_2SO_4. It follows from the assessment of the balance of iron ions performed that the amount of Fe^{2+} ions oxidized in this process was 1.46 mmol, whereby a portion of Fe^{2+} ions was contained in the solution, whereas a part, as an $Fe(OH)_3$ deposit, was contained in grains. Thus, this stage of the testing demonstrated that the drop in pH to 2.9 results in the separation of bromine into the solution.

At the next testing stage, reactants were used in a ratio to provide a 100 % oxidant excess in relation to the stoichiometric amount.

Such proportions of reactants allowed us to verify whether complete removal of Fe^{2+} ions from the solution was possible. The copolymer sample, containing

Table 1 Results of 24-h batch-wise reaction between *N*-bromosulphonamide copolymer (0.61 g, 1 mmol active bromine) and ferrous ions (200 mL 0.02 mol L^{-1} $FeSO_4$, 4 mmol), a 100 % excess of ferrous ions in relation to stoichiometry

Parameter	Value
Fe^{2+} in solution, mmol	
Before reaction	4.00
After reaction	2.54
Fe^{3+} in solution, mmol	
After reaction	0.87
Fe in sample of the resin, mmol	
Fe^{2+}	0.02
Fe^{3+}	0.58
pH	
Before reaction	3.65
After reaction	2.91
Br^- in solution, mmol	
After reaction	1.00
Active bromine in sample of the resin, mmol	
Before reaction	1.00
After reaction	0.00

1 mmol of active bromine, was contacted with 0.02 mol L^{-1} $FeSO_4$ solution containing 1 mmol Fe^{2+}. The results presented in Table 2 indicate that in the case under consideration a complete conversion of Fe^{2+} ions followed (no presence of them was found either in the post-reaction solution or in the copolymer). In the solution, the drop in Fe^{2+} ion concentration followed very quickly, which was not the case of an analogous reaction with the share of copolymers containing active chlorine [23]. The reaction was accompanied by a higher drop in pH than previously, to 2.66, and the copolymer sample changed colour from cream to brown due to the sedimentation of iron(III) hydroxides. As in the testing conducted with the reductant excess, at the beginning of the reaction, bromine passed through to the solution. In the copolymer sample analysed after the process, no presence of active bromine was found, although 50 % of its initial amount should have remained in it. The test result evidenced the usefulness of R/BrNa if the reaction is used in the processes of removal of residual amounts of Fe^{2+} from solutions under batch regime conditions.

At the next testing stage, the R/BrNa copolymer was titrated potentiometrically using the 0.02 mol L^{-1} $FeSO_4$ solution. The testing aimed to determine how high was the redox potential that accompanied the oxidation reaction of Fe^{2+} ions using R/BrNa.

As follows from the course of the curves in Fig. 2, they differ significantly from the curves plotted under the same conditions for similar copolymers containing active chlorine [23]. The formal redox potential of the system (Fig. 2a) at measurement point 5 (50 % of reductant in relation to the stoichiometric amount resulting from Eq. (4) was 878 mV at pH = 2.77, Fig. 2b). This value was significantly higher in comparison to the value of this parameter for reactions

Table 2 Results of 24-h batch-wise reaction between *N*-bromosulphonamide copolymer (0.61 g, 1 mmol active bromine) and ferrous ions (50 mL 0.02 mol L^{-1} $FeSO_4$, 1 mmol), a 100 % excess of active bromine ions in relation to stoichiometry

Time, h	Characteristic of solution/resin
	Concentration of Fe^{2+} in solution, mg L^{-1}
0	1117
1	246
3	11
5	0
24	0
	Concentration of Fe^{3+} in solution, mg L^{-1}
24	628
	Fe^{3+} in sample of the resin, mg
24	23.84
	pH
0	3.65
24	2.66
	Active bromine in sample of the resin, mmol
0	1.00
24	0.00

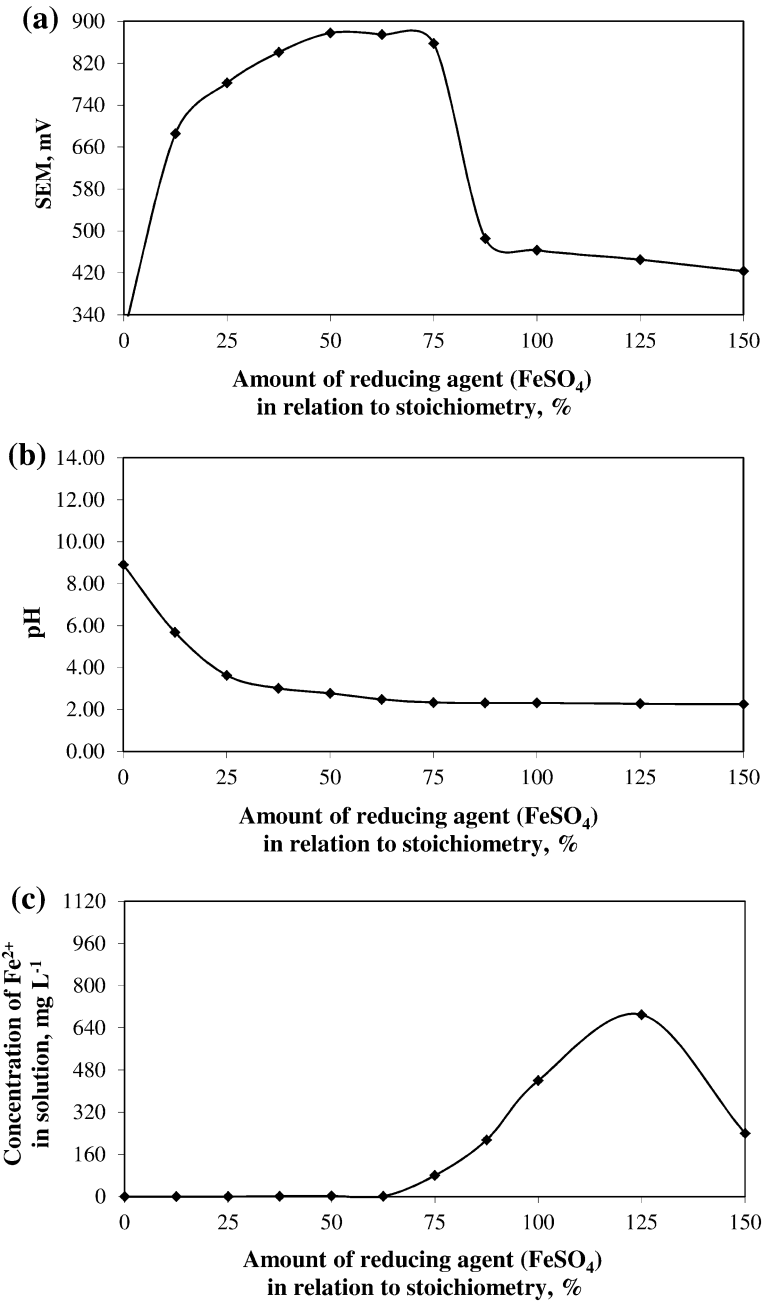


Fig. 2 **a** Redox titration curve of ferrous ions in solution during reaction of *N*-bromosulphonamide copolymer with 0.02 mol L^{-1} $FeSO_4$, **b** pH value, **c** concentration of ferrous ions in solution

conducted with the share of copolymers containing active chlorine [23]. Under the conditions described (measurement point 3 and the successive ones), R/BrNa (being in excess) oxidized Fe^{2+} ions (Fig. 2c), and the "excessive part" of active bromine passed through to the solution, which was evidenced by a change in the colour of samples from cream to brown, a pungent smell of bromine, and a relatively low pH of the media (ca. 3.0 and lower starting with point 4). What is of interest, as can be seen in Fig. 2b, c, at the first two measurement points (sample 1 without addition of the reductant and a sample with addition of 12.5 % of the reductant), the reaction progressed otherwise. It was accompanied by a rise in pH of the media, which is attributable to the partial hydrolysis of $–SO_2NBrNa$ groups and the appearance of NaOBr as a product of the reaction (similar to reaction (3)).

At the next testing stage, a bed of the R/BrNa copolymer of a ca. 5.0 g mass was used, placed in a glass column. The bed contained ca. 8.2 mmol of active bromine, and such an amount could oxidize 16.4 mmol of Fe^{2+}. As the influent, 0.0025 mol L^{-1} $FeSO_4$ solution with pH = 3.61 was used.

The results of the stationary testing showed that under the tested conditions until such a time that pH of the solution was within the range of 2.66–2.90, the oxidation of Fe^{2+} using R/BrNa was accompanied by the separation to the solution of a higher amount of bromine than resulted from the stoichiometry of the reaction (4). Therefore, the course of the column process was particularly monitored, starting at the moment of its initiation (observation of the colour of the bed and monitoring of the smell of the solution).

As follows from the curves presented in Fig. 3a, the reaction of the Fe^{2+} oxidation progressed gradually, as the inflow was supplied onto the column, and the pH of the solution did not drop below 2.98 (Fig. 3b). It turned out that under these conditions active bromine remained bound with the copolymer. As follows from the course of the curve in Fig. 3a, c, in the first fractions of the effluent (up to $V/V_0 \sim 120$, ca. 1 L of effluent) no presence of either Fe^{2+} ions or Fe^{3+} ions was found. Until that moment, such an amount of the solution was passed through the column that contained ca. 2.5 mmol of Fe^{2+} ions. Fe^{3+} ions formed as a result of the oxidation remained, at this stage, bound by sulphonic groups contained in the copolymer, and after the hydrolysis they settled partially in the bed in the form of $Fe(OH)_3$—a change in the bed colour from cream to brown was noted. The process was accompanied by a rise in the pH of the media. It should be added that during the column process, the maximum concentration of Fe^{3+} (Fig. 3c) in the effluent reached a value of 40.5 mg L^{-1}, whereas a value of 139 mg L^{-1} was theoretically achievable.

The concentration of Br^- ions, relatively high in the first fraction (2.7 mmol L^{-1}), decreased to zero in the final fractions (upon passing 4.9 L, $V/V_0 = 577$ of influent, Fig. 3d). It follows from the course of the curve that depicts the concentration of Fe^{3+} ions in the solutions received from the column that despite the completion of the Fe^{2+} oxidation reaction and with no presence of Br^- ions in the effluent, minor amounts of Fe^{3+} are present in it (<10 mg L^{-1}). This might have been caused by a partial washing out from the bed of previously precipitated iron compounds. The concentration of Fe^{2+} ions in the last fractions of the effluent was ca. 110 mg L^{-1} and was not proportional to that of Fe^{3+} ions (ca. 10 mg L^{-1}). This

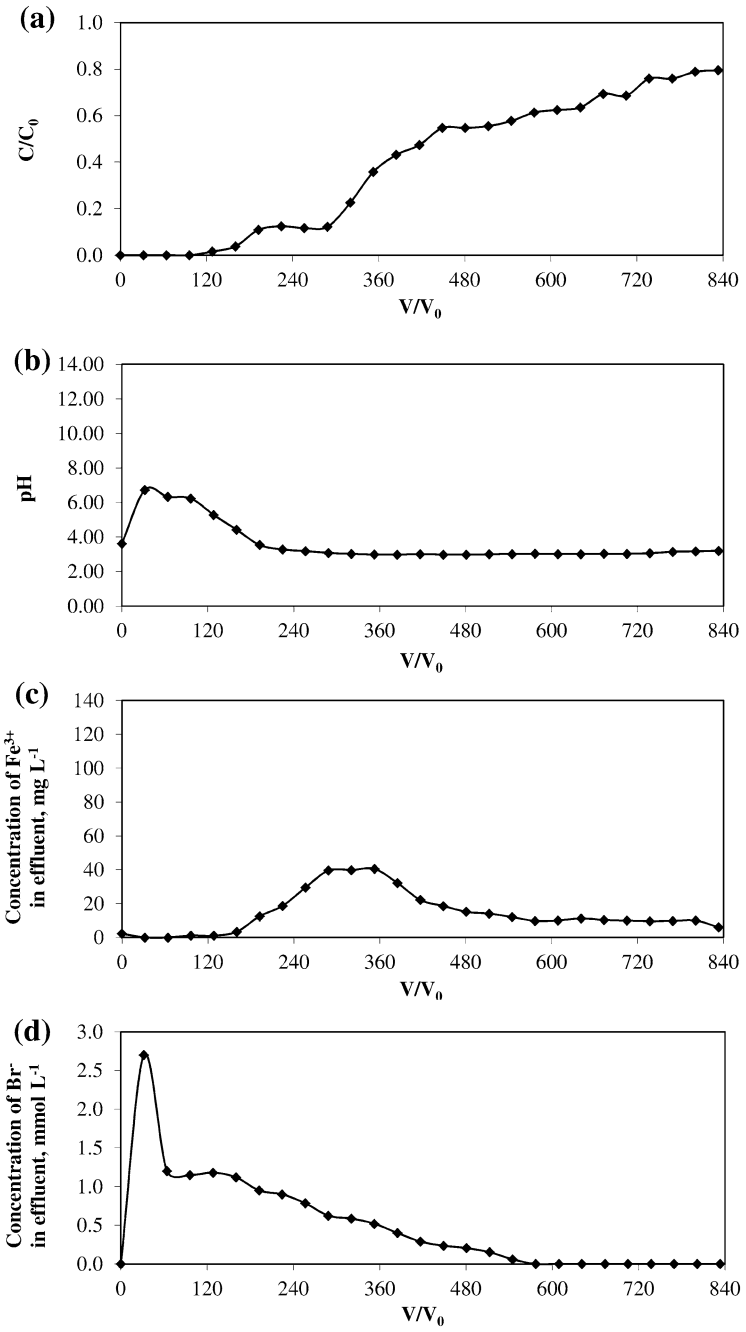


Fig. 3 **a** Ferrous ions breakthrough curve for *N*-bromosulphonamide copolymer in the column process: influx 0.0025 mol L^{-1} $FeSO_4$, flow rate 29 BV h^{-1}, **b** pH value, **c** concentration of ferric ions in effluent, **d** concentration of bromide ions in effluent

could evidence the sorption of ions or hydroxides on the surface of previously precipitated iron compounds.

The process was completed upon passing ca. 7.1 L of the solution ($C/C_0 = 0.8$, $V/V_0 = 840$), due to the fact that deposits precipitated in the bed caused problems with the flow of the solution through the column. Upon completion of the process, the bed was washed off with 1 mol L^{-1} H_2SO_4. The analysis has demonstrated the contents in the solution of 1.40 mmol of Fe^{2+} and 6.48 mmol of Fe^{3+}. The total oxidizing capacity of the bed under the tested conditions ($C/C_0 = 0.5$, $V/V_0 = 420$) was 1.78 mmol of Fe^{2+} g^{-1}, which constituted ca. 54 % of its theoretical oxidizing capacity. The column process under discussion was accomplished with a high flow rate as for the process of a redox nature. Redox reaction ran considerably slower than the ionic reaction. The slowdown of the flow of the solution through the bed would allow the use of the oxidizing capacity to a higher degree.

Conclusions

It has been demonstrated that the macroporous S/DVB copolymer containing *N*-bromosulphonamide functional groups in acidic media reacts as an oxidant in two ways. When the pH of the reaction media is below 3.0, functional groups lose bromine, which, in the reaction media, as a low molecular weight oxidant, oxidizes reducing agents present in it. Such a method of operation can be useful in processes accomplished by batches, when a powerful, heterogeneous oxidant is required; on the other hand, it is unfit for use in the column process. When the pH of reaction media is above 3.0, bromine is bound with functional groups permanently; it reacts gradually, as the solution of the reductant is supplied onto the column. It has been demonstrated that in the *N*-bromosulphonamide copolymer functional groups can appear in the hydrogen form permanently, which is not the case of the low molecular weight derivative since the free *N*-bromo-*p*-toluenesulphonamide acid is not separable from the aqueous solution. Thus, high molecular weight compounds can yield products exceeding with their structure low molecular weight compounds.

In the next work we will describe the main properties of a new polymeric reagent with *N*-bromosulphonamide functional groups in H^+ form. We will compare IR spectra and DTG/DTA charts of the copolymers with $–SO_2NBrNa$ and $–SO_2NBrH$ functional groups.

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